Effect of caries infiltrant application on shear bond strength of different adhesive systems to sound and demineralized enamel

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Abstract: Purpose: To investigate the influence of caries infiltrant application on the shear bond strength of different adhesives on sound and demineralized enamel. Materials and Methods: Sound and artificially demineralized (14 days, acidic buffer, pH 5.0) bovine enamel specimens were treated with a caries infiltrant (Icon, DMG), three different commercial adhesives (unfilled etchand- rinse adhesive: Heliobond, Ivoclar Vivadent; filled etch-and-rinse adhesive: Optibond FL, Kerr; or self-etching adhesive: iBOND Self Etch, Heraeus Kulzer) or a combination of caries infiltrant and adhesive. The shear bond strength of a nanohybrid composite was analyzed after thermocycling (5000x, 5° to 55°C) at a crosshead speed of 1 mm/min. Failure mode was inspected under a stereomicroscope at 25X magnification. Results: In both sound and demineralized enamel, the shear bond strength of the caries infiltrant was not significantly different from the etch-and-rinse adhesives, while the self-etching adhesive showed significantly lower values compared to all other groups. Pretreatment with the caries infiltrant significantly increased the bond strength of the self-etching adhesive in both substrates and of the filled etch-and-rinse adhesive in demineralized enamel. While shear bond strength was not significantly different between the two substrates, cohesive failures were more likely to occur in demineralized than sound specimens. Conclusion: The shear bond strength of the caries infiltrant was similar to the etch-and-rinse adhesives. The caries infiltrant did not impair bonding to sound or demineralized enamel, and even increased adhesion of the selfetching agent.

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Abstract

Purpose: The purpose of this study was to investigate the influence of caries infiltrant application on the shear bond strength of different adhesives on sound and demineralized enamel.

Materials and Methods: Sound and artificially demineralized (14 d, acidic buffer, pH 5.0) bovine enamel specimens were treated with a caries infiltrant (Icon, DMG), three different commercial adhesives (unfilled etch&rinse adhesive: Heliobond, Ivoclar Vivadent; filled etch&rinse adhesive: Optibond FL, Kerr; or self-etching adhesive: iBOND Self Etch, Heraus Kulzer) or a combination of caries infiltrant and adhesive. Shear bond strength of a nanohybrid-composite was analysed after thermo-cycling (5000x, 5^{o} to 55^{o}C) at a crosshead speed of 1 mm/min. Failure mode was inspected under stereomicroscope at 25× magnification.

Results: In both sound and demineralized enamel, shear bond strength of the caries infiltrant was not significantly different from the etch&rinse adhesives, while the self-etching adhesive showed significantly lower values compared to all other groups. Pretreatment with the caries infiltrant significantly increased the bonding strength of the self-etching adhesive in both substrates and of the filled etch&rinse adhesive in demineralized enamel. While shear bond strength was not significantly different between both substrates, cohesive failures were more likely to occur in demineralized than sound specimens.

Conclusion: The shear bond strength of the caries infiltrant was similar to the etch&rinse adhesives. The caries infiltrant did not impair bonding to sound and demineralized enamel, and even increased adhesion of the self-etching agent.

Keywords

enamel, demineralization, caries infiltrant, adhesive, shear bond strength

Introduction

Caries infiltration techniques have been increasingly studied in the last years. Surface conditioning of enamel and composition of the infiltrant have been systematically tested in various in vitro studies in order to improve the penetration of the infiltrant resin in the carious lesion.[12,15,21,22,26] It is well known yet that infiltrants with a low viscosity and a high penetration coefficient are able to penetrate natural carious lesions almost completely.[13,15] Clinical data are scarce – not least as the commercial product was introduced only in 2009 - but show that the progression of the carious lesion can be slowed down by the application of the infiltrant resin.[24,25] Besides, resin infiltrants were also used to improve the visual appearance of white spot lesions.[18,23,27]

To extend the range of application, it was recently questioned whether a caries infiltrant can be also used for conditioning of enamel prior to placement of a composite filling.[33] This issue is of clinical relevance in cases of initial, but cavitated lesions, which might require both the restoration of cavitated and the infiltration of demineralized areas. Ideally, infiltration of the demineralized parts could be done in the same step as the conditioning of enamel and dentin. In a previous study it was shown that the adhesion of a flowable composite to sound and demineralized enamel could be achieved to the same extent by a caries infiltrant and a conventional bonding agent. Moreover, the application of the infiltrant resin prior to bonding application did not impair the shear bond strength of the bonding agent.[33] These promising preliminary results require further investigation of bonding techniques where caries infiltration can be combined with adhesive conditioning. Therefore, this study aimed to compare the adhesive performance of a caries infiltrant with different adhesives (unfilled etch&rinse adhesive, filled etch&rinse adhesive and self-etching adhesive) on sound and demineralized enamel, and to analyze if the application of a caries infiltrant influences the shear bond strength of the respective adhesive when applied successively. In contrast to the preliminary study, shear bond strength was assessed after aging of the specimens, as TEGDMA-based resins, such as the caries infiltrant, exhibit a higher hydrophilicity and thus, higher susceptibility to degradation compared to resins containing less TEGDMA.[30] The null hypotheses of the present study were: 1) The shear bond strength of the caries infiltrant system on sound and demineralized enamel is not significantly different from the conventional adhesives tested and 2) pretreatment with the caries infiltrant does not impair bonding strength of the different adhesives.

Material and Methods

Specimen preparation

Cylindric enamel specimens (6.6 mm in diameter, n = 210) were prepared from the crowns of freshly extracted, non-damaged bovine incisors, which were stored in 0.9% NaCl solution[8] until used. The specimens were then embedded in chemically cured acrylic resin (ScandiQuick, ScanDia, Hagen, Germany) and ground flat with 400-grit SiC paper (Buehler, Lake Bluff, USA).

For demineralization, half of the specimens (n = 105) were stored for 14 days at 37°C in a acid buffer containing 3 mM $CaCl_2 \cdot 2\,H_2O$, 3 mM KH_2PO_4, 50 mM acetic acid, 6 μM MHDP per 5 liters, and KOH to adjust the initial pH to pH 5.0.[5] The solution was renewed each second day to keep the pH constant. The artificial lesions created by storage in the acid buffer were proven to exhibit the typical histological structure of caries lesions.[1,10]

Bonding procedure

Sound and demineralized specimens were randomly divided into seven groups (each n = 15), and the enamel surface (6.6 mm in diameter) was treated with either the caries infiltrant, one of the adhesives or a combination of the caries infiltrant and an adhesive. Contamination of the embedding resin with the caries infiltrant and/or the adhesives was avoided. The compositions of the conventional adhesives and the caries infiltrant system according to its manufacturers` instructions are listed in Table 1.

Group 1: Caries infiltrant

The enamel surface was etched with 15% hydrochloric acid gel (Icon Etch, DMG, Hamburg, Germany) for 2 min and then rinsed with water for 30 s. The surface was dried with ethanol (Icon Dry, DMG, Hamburg, Germany), applied for 30 s. Then, the low-viscosity infiltrant resin (Icon Infiltrant, DMG, Hamburg, Germany) was applied on the surface for 3 min with a sponge applicator. The infiltrant was light cured for 40 s at 800 W/cm^2 (bluephase, IvoclarVivadent, Schaan, Liechtenstein). After light curing, the infiltrant was applied again for 1 min and light cured for 40 s.

Group 2: Unfilled etch&rinse adheisve

The enamel surface was etched with 37% phosphoric acid gel (Total Etch, Ivoclar Vivadent, Schaan, Liechtenstein) for 30 s and then rinsed with water for 30 s. After drying, the adhesive (Heliobond, IvoclarVivadent, Schaan, Liechtenstein) was applied in a thin layer and light-cured for 20 s.

Group 3: Filled etch&rinse adhesive

Enamel was etched with 37% phosphoric acid (Total Etch, Ivoclar Vivadent, Schaan, Liechtenstein) prior to application of an etch&rinse adhesive (Optibond FL, Kerr, California, USA). The primer was applied for 15 s and gently air-dried for 5 s. Then, the adhesive was applied for 15 s, gently air-thinned and light-cured for 20 s.

Group 4: Self-etching adhesive

The self-etching adhesive (iBOND Self Etch, Heraeus Kulzer, Hanau, Germany) was applied on the enamel surface for 20 s, gently air-dried for 5 s and light-cured for 20 s.

Groups 5 - 7: Caries infiltrant + adhesives

Samples were first treated with the caries infiltrant as described in group 1. Then, the respective etch&rinse adhesive (Heliobond: group 5; Optibond FL: group 6) or self-etching adhesive (iBOND Self Etch: group 7) was applied and light-cured as described above.

A nano-hybrid composite (TetricEvoCeram, Ivoclar Vivadent, Schaan, Liechtenstein) was adhered to the enamel surface by means of a transparent plastic hollow cylinder with an inner diameter of 3 mm as described in detail previously (Figure 1).[28] The composite was packed against the surface in a 2 mm thick increment, which was then light-cured for 60 s. Light intensity was higher than 800 mW/cm^2 as confirmed by a radiometer (Optilux Model 100, SDS Kerr Danbury, USA) after each 15 specimens. Bonding procedures were carried out by the same operator throughout the experiments.
The specimens were thermo-cycled 5000 times between 5° and 55°C (Willytec, Gräfelfing, Germany) prior to shear bond strength testing.[19,20]

Shear bond test

Shear bond test was performed with an universal testing machine (Z010, Zwick, Ulm, Germany). A shear force was applied to the adhesive interface through a chisel-shaped loading device at a crosshead speed of 1 mm/min parallel to the enamel surface. Load at fracture was recorded and shear bond strength was calculated by a software (TestXpert 11.02, Zwick, Ulm, Germany) using the load at failure and the adhesive area.

The debonded area was examined for failure mode analysis with a stereomicroscope at 25x magnification (M3B, Wild, Heerbrugg, Switzerland). Failure mode was considered as adhesive, if it occurred at the interface and as cohesive if at least parts of enamel or composite were affected.

Statistical analysis

Descriptive statistics for shear bond strength (mean ± standard deviation, 95% confidence intervals (CI)) were computed. As bond strength data were normally distributed (except group 1/demineralised samples, Kolmogorov-Smirnov test) and had homogeneous group variances (Bartlett test), data were analysed by two-way analysis of variance (ANOVA), factors being the substrate (sound or demineralized enamel) and the group (bonding procedure). Within sound and demineralized enamel, significant differences between the groups were analysed by one-way ANOVA and Scheffe`s post-hoc tests.

Relative frequencies of cohesive failures in each group were calculated at 95% CI. Frequency of cohesive failures was analysed by logistic regression using the substrate and the group as factors. The level of significance was set at $p \leq 0.05$.

Results

Shear bond strength values of sound and demineralized enamel groups and the respective failure modes are presented in Tables 2 and 3.

Two-way ANOVA found the bonding procedure ($p < 0.0001$) but not the enamel substrate ($p = 0.541$) to be significant with respect to shear bond strength. No significant interaction between the factors was detected ($p = 0.051$). Within sound and demineralized enamel, one-way analysis showed significant differences between the groups.

In both substrates, the self-etching adhesive exhibited significantly lower shear bond strength values than all other groups. Pretreatment with caries infiltrant increased shear bond strength of the self-etching adhesive in sound and demineralized enamel to the level of the etch&rinse adhesives. While the shear bond strength of the unfilled etch&rinse adhesive was affected by the application of the caries infiltrant neither in sound nor in demineralized enamel, the adhesive performance of the filled etch&rinse adhesive was significantly increased in demineralized samples.

Cohesive failures occurred only in enamel. Frequency of cohesive failures was affected by the kind of substrate but not by the bonding procedure. Thus, more cohesive failures were observed in demineralized compared to sound enamel.

Discussion

This in vitro study showed that the adhesive performance of a caries infiltrant was similar to conventional adhesives even under aging conditions, and that pretreatment with the caries infiltrant did not impair bonding strength of the adhesives but even increased the shear bond strength of iBOND Self Etch. Thus, the null hypotheses of the study were rejected.

Shear bond strength was tested on sound and artificially demineralized samples as the enamel margins after cavity preparation might comprise both sound and demineralized areas. Although the artificial lesions used in this study were shown to have a similar histological structure compared to natural white spot lesions, the surface layer is thicker in natural (~ 40 to 50 µm and) compared to artificial (~ 20 µm) lesions.[2,10] While natural white spot lesions require hydrochloric acid etching to remove this surface layer completely.[12] and allow for successful penetration of an infiltrant, it might be assumed that hydrochloric acid etching of artificial lesions does not only remove the surface layer but induce also the breakdown of parts of the lesion. Thus, artificial enamel lesions used for infiltration tests were usually etched for 5 s with 20% or 37% phosphoric acid,[6,14,17,24] attempting only the removal of the compact surface layer. However, in a recent study,[1] it was shown that etching with 37% phosphoric acid was unable to remove the 25 µm thick surface layer of artificial lesions. Cross-sectional scanning electron microscopic images of artificial white spot lesions etched with phosphoric acid showed a poorly dissolved surface layer remaining. In contrast, hydrochloric acid etching for 120 s removed the surface layer of the artificial lesions completely, without destroying the underlying structures.[1] Therefore, in the present study, enamel specimens prepared for infiltration were etched with hydrochloric acid for 120 s as recommended by the manufacturer of the commercial product, while the etch&rinse adhesives were etched by phosphoric acid when not combined with the infiltrant system.

As shown previously for non-altered specimens,[33] the adhesive performance of the caries infiltrant is in the range of the etch&rinse adhesives also after aging of the specimens. The high amount of TEGDMA in the resin infiltrant promotes the penetration of the resin,[21] but might also increase the susceptibility to degradation compared to resins containing less TEGDMA. Moreover, infiltrated lesions often exhibit inhomogenities, probably as a result of polymerization shrinkage and polymerization stress of the resin,[22] which might increase the risk of leakage and, thus, might affect bonding strength. However, the results of the present study indicate that the adhesion of the TEGDMA-based resin infiltrant is not affected in a way that the shear bond strength is significantly reduced compared to the adhesives containing less TEGDMA.

The shear bond strength of the caries infiltrant was compared to Heliobond, Optibond FL and iBOND Self Etch, which were chosen as representatives for an unfilled and filled etch&rinse adhesive, respectively, and a self-etching adhesive. In accordance to previous studies,[3,31] the self-etching adhesive showed significantly lower shear bond strength values compared to the etch&rinse adhesives, which require phosphoric acid etching of enamel prior to their application. Gregoire and Ahmed[7] showed that the etching efficacy of iBOND Self Etch on ground enamel was significantly less intensive compared to phosphoric acid resulting in a less deep demineralization and more irregular etching pattern than conventional etching.[7] Moreover, according to Mueller et al,[17]acidic monomers of self-etching adhesives were unable to erode the surface layer of the artificial lesions adequately to allow for penetration of the adhesive. As self-etching adhesives were shown to benefit from pre-etching with phosphoric acid,[32] a similar effect can be assumed for etching with hydrochloric acid in groups where iBOND Self Etch was combined with the caries infiltrant. Hydrochloric acid might induce a honeycomb etching pattern in sound enamel[9] and remove the surface layer of the artificial lesions, both resulting in a better penetration of the adhesive.

Besides the kind of etching, bond strength in the different groups might be also affected by the penetration capability of the adhesives. Infiltrants containing TEGDMA and HEMA were shown to have a better penetration coefficient than resins containing large amounts of BISGMA or UDMA. The penetration coefficient might be also affected by the solvent. It was discussed that the addition of ethanol enhances the penetration coefficient to some extent, while acetone seemed to decrease penetration.[11,21] Therefore, it can be assumed that the penetration of the self-etching adhesive containing UDMA and acetone is reduced compared to the adhesives containing TEGDMA or HEMA and ethanol. Besides the monomer composition, the penetration is also affected by the filler content. While the penetration behaviour might be reduced by the addition of fillers, the enhanced physical properties of filled compared to unfilled adhesives might improve the bonding strength.[16] Thus, in the present study, no significant differences between the unfilled and the filled etch&rinse adhesives were observed.

Although conventional adhesives might penetrate demineralized enamel less deep than infiltrants, the penetration might be more homogenous compared to the infiltration. As a result, the adhesion capability of the complete, but inhomogeneous infiltration and incomplete, but homogenous penetration of the etch&rinse adhesives are equally effective.

The pretreatment with the TEGDMA-containing infiltrant probably results in a thick oxygen-inhibited layer[29], which allows the chemical connection between the infiltrant and the adhesive or composite monomers, respectively. However, the pretreatment with the caries infiltrant increased the adhesion of Heliobond and Optibond FL only slightly, but mostly not significant. In contrast, adhesion of the self-etching adhesive could be enhanced significantly by the infiltrant, probably as a result of a deeper penetration of the infiltrant.

Generally, cohesive failures were more often seen in demineralized enamel. As shear bond strength did not vary significantly between sound and demineralized enamel, this effect might be partly explained by the brittleness of demineralized enamel, which is more likely to fracture under stress. Provided that the self-etching adhesive penetrated less deep into demineralized enamel compared to the other groups, this effect might account for its high number of cohesive failures in demineralized enamel. In contrast, cohesive failures were observed less frequently when the self-etching adhesive was combined with the caries infiltrant, potentially as a result of a better stress distribution and stabilization of the demineralized enamel by the infiltrant.

In contrast to a previous study by Wiegand et al,[33] absolute shear bond strength values were distinctly higher in the present study, which can be explained by different elastic moduli of the composites used (flowable composite vs nano-hybrid composite)[4] and the influence of the different operators.[31]

In conclusion, the shear bond strength of the caries infiltrant was similar to the etch&rinse adhesive. The caries infiltrant did not impair bonding to sound and demineralised enamel, and even increased adhesion of the self-etching agent.

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Table 1 Composition of the caries infiltrant and the adhesive systems

Product	Composition	Batch number	Manufacturer
Icon	Icon-Etch: hydrochloric acid, pyrogenic silicic acid, surface-active substances	635703	DMG, Hamburg, Germany
	Icon-Dry: 99% ethanol	633314	
	Icon-Infiltrant: TEGDMA-based resin matrix, initiators, additives	633139	
Heliobond	< 60% Bis-GMA,< 40% TEGDMA, stabilizers and catalysts	N26864	Ivoclar Vivadent, Schaan, Liechtenstein
Optibond FL	Primer: HEMA, ethanol, GPDM, MMEP, water, CQ, BHT	3463213	Kerr, Orange, California, USA
	Adhesive: Bis-GMA, HEMA, GDMA, CQ, ODMAB, approximately 48wt% filled	3486699	
iBOND Self Etch	UDMA, 4-META, glutaraldehyde, acetone, water, photo-initiators, stabilizers	010104	Heraeus, Hanau, Germany

Bis-GMA=bisphenol A diglycidyl methacrylate, TEGDMA=triethylene glycol dimethacrylate, HEMA=2-hydroxyl methacrylate, GPDM=glycerol phosphate dimethacrylate, MMEP=mono-2-methacryloyloxyethyl phthalate, CQ= camphorquinone, BHT=butylhydroxytoluene, GDMA=glycerol dimethacrylate, ODMAB=2-(ethylhexyl)-4-(dimethylamino)benzoate, UDMA=urethane dimethacrylate, 4-META=4-mathacryloyloxyethyl trimellitate anhydride.

Table 2 Shear bond strength (MPa, mean ± standard deviation, 95% confidence intervals (CI)) of sound and demineralized enamel groups.

Enamel	Groups	Mean(MPa)±SD	95% CI	
Sound	Icon	20.4 ± 4.5[a]	17.9	22.9
	Heliobond	21.6 ± 5.4[a]	18.5	24.6
	Icon + Heliobond	22.5 ± 3.6[a]	20.5	24.4
	Optibond FL	21.0 ± 6.2[a]	17.5	24.5
	Icon + Optibond FL	21.6 ± 5.3[a]	18.7	24.5
	iBOND Self Etch	13.4 ± 6.1[b]	10.0	16.8
	Icon + iBOND Self Etch	21.8 ± 4.3[a]	19.4	24.2
Demineralized	Icon	22.7 ± 5.4[ab]	19.7	25.7
	Heliobond	19.9 ± 5.3[ab]	16.9	22.8
	Icon + Heliobond	23.0 ± 4.7[ab]	20.4	25.6
	Optibond FL	18.6 ± 4.8[b]	15.9	21.3
	Icon + Optibond FL	25.6 ± 5.7[a]	22.5	28.6
	iBOND Self Etch	10.9 ± 4.7[c]	8.3	13.5
	Icon + iBOND Self Etch	24.5 ± 2.9[ab]	22.9	26.2
Within sound or demineralized groups, shear bond values which were not significantly different, are marked with the same letter.				

Table 3 Adhesive and cohesive failures and relative frequency of cohesive failures (95% CI) in the different groups

Cohesive failures occurred only in enamel but not in the composite

Enamel	Groups	Number of failure		Relative frequency (%) of cohesive failures (95% CI)
		adhesive	cohesive	
Sound	Icon	12	3	20.0 (4.3; 48.1)
	Heliobond	13	2	13.3 (1.7; 40.5)
	Icon + Heliobond	10	5	33.3 (11.8; 61.6)
	Optibond FL	11	4	26.7 (7.8; 55.1)
	Icon + Optibond FL	13	2	13.3 (1.7; 40.5)
	iBOND Self Etch	14	1	6.7 (0.2; 32.0)
	Icon + iBOND Self Etch	14	1	6.7 (0.2; 32.0)
Demineralized	Icon	8	7	46.7 (21.3; 73.4)
	Heliobond	11	4	26.7 (7.8; 55.1)
	Icon + Heliobond	12	3	20.0 (4.3; 48.1)
	Optibond FL	5	10	66.7 (38.4; 88.2)
	Icon + Optibond FL	10	5	33.3 (11.8; 61.6)
	iBOND Self Etch	2	13	86.7 (59.5; 98.3)
	Icon + iBOND Self Etch	11	4	26.7 (7.8; 55.1)

Figure 1 Specimen preparation and shear bond strength testing

Detail view of the specimen clamp holding an embedded enamel cylinder and the fixation of the acrylic cylinder already filled with composite (A)

Specimen loaded in the universal testing machine (B)